DERWENT-ACC-NO:

1994-212990

DERWENT-WEEK:

199426

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TITLE:

Electroconductive resin paste for semiconductor device - comprises epoxy! resin cpd, hardener, electroconductive

filler and opt diluent to mitigate thermal pressure

PATENT-ASSIGNEE: HITACHI CHEM CO LTD[HITB]

PRIORITY-DATA: 1992JP-0303684 (November 13, 1992)

PATENT-FAMILY:

PUB-NO

PUB-DATE

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JP 06151480 A

May 31, 1994

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APPLICATION-DATA:

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November 13, 1992

INT-CL (IPC): C08G059/42, C08L063/00, C09J163/00, H01L021/52

ABSTRACTED-PUB-NO: JP 06151480A

BASIC-ABSTRACT:

Resin paste comprises (A) epoxy resin prepd by reacting epoxy resin contg at least 2 epoxy gps, an aliphatic monocarboxylic acid and <u>polydimethylsiloxane</u> cpd contg COOH, (B) a hardener and (C) an electroconductive filler and opt (D) a diluent.

Also claimed a semiconductor device bonded with the electroconductive resin paste and then sealing the device.

The starting epoxy resin is eg glycidyl ether of bisphenol A, -AD, -F or S, phenol novolak or cresol novolak or polyglycidyl ether of polyhydric phenol. The aliphatic monocarboxylic acid is pref (6-19C) unsatd branched monocarboxylic acid (eg caprylic acid or oleic acid).

The <u>polydimethylsiloxane</u> cpd is eg XC-96-A1768 or XC-96-A 2400 (RTM: YSIL) or PS5g2 (RTM: CHCC) or <u>polydimethylsiloxane</u> cpd of T-structure (eg PS402 RTM: CHCC).

(A) is prepd by reacting such epoxy resin, aliphatic monocarboxylic acid and

<u>polydimethylsiloxane</u> cpd at 100-190 deg C opt in the presence of a catalyst (eg a tertiary amine, imidazole or triphenyl phosphate) in the solvent.

- (B) is eg phenol novolak, phenol aralkyl resin, dicyandiamide, dibasic acid hydrazide, imidazole, organic borate, a tert amine or its salt, metal acetylacetonate and used in a wt ratio/(A): 0.001-2.0. (C) is eg <u>flake</u>, dendrite or sphere of Au, <u>Ag</u>, Gu, Ni, <u>Al</u>, Fe or stainless steel and used in a wt ratio/((A)+(B)) of 1.00-10.0.
- (D) is eg an organic solvent (eg butyl cellosolve, butyl cellosolve acetate, carbitol, carbitol acetate or ethylene glycol diethyl ether) or a reactive diluent (eg phenylglycidyl ether or p-t-butylphenylglycidyl ether) and used in a wt ratio/((A)+(B)) of 0-80.

The paste is blended opt with an adhesion improver (eg silane coupling agent or Ti coupling agent), a wettability improver (eg a nonionic surfactant or F-contg surfactant) or defoaming agent.

ADVANTAGE - The resin <u>paste mitigates thermal</u> stress caused by the difference in the thermal expansion coeffts of a semiconductor element, lead frame and base board. It prevents failure in a semiconductor element due to the chip crack and the strain in the heat-treatment of chip and has high workability, hardenability and adhesion to be used for a large sized chip.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: ELECTROCONDUCTING RESIN PASTE SEMICONDUCTOR DEVICE COMPRISE

POLYEPOXIDE RESIN COMPOUND HARDEN ELECTROCONDUCTING FILL OPTION DILUTE MITIGATE THERMAL PRESSURE

DERWENT-CLASS: A21 A26 A85 L03 U11

CPI-CODES: A05-A01E2; A06-A00E2; A08-M09A; A09-A03; A10-E07; A12-E04; A12-E07C; L04-C20A;

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UNLINKED-DERWENT-REGISTRY-NUMBERS: 0973U; 1193U ; 1264U ; 5007U ; 5319U ; 5333U

ENHANCED-POLYMER-INDEXING:

Polymer Index [1.1]

017; G1570*R G1558 D01 D11 D10 D23 D22 D31 D42 D50 D69 D83 F47
7A G1274*R G1092 D18 F33 F30; R00470 G1161 G1150 G1149 G1092 D01
D11 D10 D19 D18 D32 D50 D93 F32 F30; R12487 G1207 G1150 G1149 G1092
D01 D11 D10 D19 D18 D32 D50 D93 F32 F30; R00473 G1230 G1150 G1149
G1092 D01 D19 D18 D32 D50 D92 F32 F30 F61; P0464*R D01 D22 D42

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Notes:

- 1. Untranslatable words are replaced with asterisks (****).
- 2. Texts in the figures are not translated and shown as it is.

Translated: 06:33:42 JST 09/09/2006

Dictionary: Last updated 08/25/2006 / Priority: 1. Chemistry / 2. Natural sciences / 3. Manufacturing/Quality

CLAIMS

[Claim(s)]

[Claim 1] (A) In the conductive resin paste which contains the (D) diluent an epoxy resin, the (B) curing agent, (C) conductivity filler, and if needed The conductive resin paste whose epoxy resin (A) is the resultant of the poly dimethylsiloxane compound which has the epoxy resin, the aliphatic monocarboxylic acid, and the carboxyl group which have two or more epoxy groups in 1 molecule.

[Claim 2] The conductive resin paste according to claim 1 whose curing agent is a phenol resin.

[Claim 3] The semiconductor device closed after pasting up a semiconductor device and a substrate with a conductive resin paste according to claim 1.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to a conductive resin paste, a suitable conductive resin [to paste up semiconductor devices, such as IC and LSI, on substrates such as a lead frame, a ceramic patchboard, and a glass epoxy patchboard, in more detail] paste, and the semiconductor device using this.

[0002]

[Description of the Prior Art] The conductive resin paste is mainly used for adhesion (die bonding) with semiconductor devices, such as IC and LSI, and substrates, such as a lead frame, in recent years. However, in connection with rapid progress of semiconductor industry, the problem of the thermal stress by the difference in the coefficient of thermal expansion of a large-sized semiconductor device and substrates, such as a lead frame, is actualizing, and it is this. How to add (1) carboxylic-acid end polybutadiene to a conductive resin paste as a means

to solve (JP,S62-199669,A), (2) The method (JP,S63-10104,A) of adding the dimethylsiloxane compound which has the method (JP,S63-161015,A), (3) amino groups, or the hydroxyl group which adds epoxidation polybutadiene etc. is performed. However, since the molecular weight of a carboxylic acid end polybutadiene modified epoxy resin is large by the method of (1), It is difficult to harden by the method of (2), bad [the spreading workability of a conductive resin paste], for a short time, since the hardenability of epoxidation polybutadiene is low, and it is inferior to productivity. Furthermore, by the method of (3), since the compatibility of a dimethylsiloxane compound and an epoxy resin is bad, there is a fault, such as causing separation, during preservation of conductive paste.

[0003]

[Problem(s) to be Solved by the Invention] This invention offers the semiconductor device using a conductive resin paste and this excellent in the relaxation effect of thermal stress while it solves the problem of the aforementioned conventional technology and is excellent in workability, hardenability, and compatibility.

[0004]

[Means for Solving the Problem] In the conductive resin paste with which this invention contains the (D) diluent the (A) epoxy resin, the (B) curing agent, (C) conductivity filler, and if needed The epoxy resin with which an epoxy resin (A) has two or more epoxy groups in 1 molecule, After pasting up a semiconductor device and a substrate with the conductive resin paste which is the resultant of the poly dimethylsiloxane compound which has aliphatic monocarboxylic acid and a carboxyl group, and this conductive resin paste, it is related with the semiconductor device to close.

[0005] As an epoxy resin which has two or more epoxy groups in 1 molecule used for this invention (1) For example, bisphenol A, such as trade name Epicoat 828 by an oil recovery shell epoxy company, Epicoat 1001, the trade name R-1710 by a Mitsui petrochemical company, Tohto Kasei Co., Ltd. make trade name YDF-170, and Nippon Kayaku Co., Ltd. make trade name EBPS-300, bisphenol A D, diglycidyl ether, such as bisphenol F and Bisphenol S, and (2) -- for example, Poly glycidyl ether, such as phenolic novolacs, such as Dow Chemical Co. make trade name DEN-438, Tohto Kasei Co., Ltd. make trade name YDCN-702, and YDCN-704, and cresol novolak resin, and (3), for example, the following constitutional formula, (I)

[Formula 1]

They are the trade name 1032H by an oil recovery shell epoxy company expressed with (n is 0 or a positive integer), Nippon Kayaku Co., Ltd. make EPPN-501H, Dow Chemical Co. make trade name TACTIX-742, and the following constitutional formula (II).

[Formula 2]

Mitsui petrochemical company make trade name VG-3101 come out of and expressed, the following constitutional formula (III)

[Formula 3]

(Polyhydric phenol poly glycidyl ether and (4), for example, the following constitutional formulas, (IV), such as the trade name 1031S by an oil recovery shell epoxy company come out of and expressed)

[Formula 4]

Dainippon Ink & Chemicals, Inc. make trade name HP-4032 come out of and expressed, the following constitutional formula (V)

[Formula 5]

the poly glycidyl ether which has naphthalene frames, such as Dainippon Ink & Chemicals, Inc. make trade name EXA-4300 expressed with (m is one or more integers), and (5) -- for example, Cycloaliphatic epoxy resin, such as UCC company make trade name ERL-4201, 4221, and Daicel Chemical Industries, Ltd. make trade name EHPE-3150, etc. is mentioned. [0006] As aliphatic monocarboxylic acid used for this invention For example, acetic acid. propionic acid, crotonic acid, butanoic acid, tiglic acid, pivalate, Valeric acid, sorbic acid, caproic acid, 2-ethyl butanoic acid, tert-butyl acetic acid, Isocaproic acid, oenanthic acid, a heptene acid, a 2-octene acid, caprylic acid, 2-ethyl hexanoic acid, 2-propyl pentanoic acid, 2nonene acid, nonoic acid, 2-decene acid, capric acid, undecylenic acid, undecylic acid, lauric acid, A tridecane acid, myristic acid, a pentadecane acid, palmitic acid, 2-hexyl decanoic acid, Stearin acid, isostearic acid, oleic acid, elaidic acid, heptadecanoic acid, heptadecenoic acid, linolic acid, linolenic acid, parinaric acid, a nonadecane acid, arachidic acid, etc. are mentioned. The aliphatic monocarboxylic acid which has a double bond by the shape of a side chain of a carbon number 6-18 from compatibility with the reduction effect of thermal stress, a solvent, or other resin and a point of the reduction in the viscosity of conductive paste is [among these] desirable.

[0007] As a poly dimethylsiloxane compound which has the carboxyl group used for this invention, there is no restriction in particular, for example, it is the following general formula (VI).

[Formula 6]

as the poly dimethylsiloxane compound and trade name which are expressed with (m is one or more integers and n is 0 or one or more integers) -- XC-96-A1768 and XC96-A -- 2400 (made by Toshiba Silicone) PS562 (made by Chisso Corp.)

The following general formula (VII)

[Formula 7]

$$\begin{array}{c} \text{CH}_{3} \\ \text{CH}_{4} \\ \text{CH}_{5} \\$$

As T structure poly dimethylsiloxane compound and the trade name which are expressed with (p, m, and n are one or more integers), it is PS402 (made by Chisso Corp.), for example. The following general formula (VIII)

[Formula 8]

As T structure poly dimethylsiloxane compound and the trade name which are expressed with (p, m, and n are one or more integers), PS409 (made by Chisso Corp.) etc. is raised, for example. The poly dimethylsiloxane compound which has two carboxyl groups in 1 molecule

from a point of absorption of thermal stress is desirable.

[0008] In this invention, the resultant of the poly dimethylsiloxane compound which has the epoxy resin, the aliphatic monocarboxylic acid, and the carboxyl group which have two or more epoxy groups in 1 molecule as an epoxy resin (A) is used. It is desirable to use the EPOSHIKI resin which has three or more epoxy groups into 1 molecule from the cure reaction nature of the resultant included during a conductive resin paste.

[0009] [the reaction of the poly dimethylsiloxane compound which has the epoxy resin, the aliphatic monocarboxylic acid, and the carboxyl group which have two or more EPOSHIKI machines in 1 molecule] A catalyst can be added under existence of a solvent if needed, and it can carry out by carrying out definite-period-of-time churning at the temperature of 100-190 degrees C. Pursuit of a reaction is possible by titrating the acid number by the alcoholic solution of potassium hydroxide. When the third class amines, imidazole derivatives, triphenyl phosphine, etc. are used and it is considered as conductive paste as a solvent as a catalyst added if needed, the solvent which can be used as it is is desirable.

[0010] [the blending ratio of coal of a poly dimethylsiloxane compound which has the epoxy resin, the aliphatic monocarboxylic acid, and the carboxyl group which have two or more epoxy groups in 1 molecule] It is desirable to make into a 0.01 in all to 0.40 carboxylic acid equivalent aliphatic monocarboxylic acid and the poly dimethylsiloxane which has a carboxyl group from points, such as hardenability and compatibility, to 1 weight per epoxy equivalent of an epoxy resin. [the blending ratio of coal of poly dimethylsiloxane which has aliphatic monocarboxylic acid and a carboxyl group] It is desirable to make into the rate of a 0.01 - 0.50 carboxylic-acid equivalent the poly dimethylsiloxane which has a carboxyl group from points, such as compatibility, to an aliphatic-monocarboxylic-acid 1 carboxylic-acid equivalent. [0011] In this invention, it is desirable to use together the epoxy resin which has two or more epoxy groups as an epoxy resin (A) in 1 molecule described above besides the abovementioned resultant from the point which raises the hardenability of a conductive resin paste. [0012] As a curing agent (B) used for this invention, the phenolic novolac of Meiwa Chemicals trade name H-1 grade, Phenol aralkyl resin of Mitsui Toatsu Chemicals, Inc. make trade name XL-225 grade, Dicyandiamide of Japanese carbide company make trade name SP-10 grade, the trade name ADH by a Japanese hydrazine industrial company, Dibasic acid dihydrazide, such as IDH and SDH, Shikoku Chemicals trade name cure ZORU 2MZ, 2E4MZ, 2E4 MZ-CN, 2E4 MZ-AZINE, C11Z, C11 Z-CN, C11 Z-CNS, C17Z, 2PZ, 2P4MZ, 2 PZ-CN, 2 PZ-CNS, 2 PZ-OK, 2 MA-OK, 2PHZ, Imidazole derivatives, such as 2P4MHZ, the Hokko Chem trade name TPPK, EMZ-K, San Apro trade name U-Cat The organic boron salt of the 5002nd grade, the San Apro trade name DBU, U-Cat Acetylacetone metal salt, such as the third class amines, such as 102, 106, 830, 340, benzyldimethylamine, tree 2, 4, and 6-dimethylamino methyl phenol, and the salt acetylacetone aluminium, acetylacetone magnesium, etc. is

mentioned. These are independent or are used combining two or more sorts. Although the amount of the curing agent used is made into sufficient quantity to maintain the working life of a conductive resin paste more than fixed, and hold practical hardenability, it is usually the range of a 0.1 - 200 weight part to an epoxy resin (A) 100 weight part.

[0013] Although the minute powder of conductive metals, such as gold, silver, copper, nickel, an aluminium, iron, and stainless steel, etc. is mentioned as a conductive filler (C) used for this invention, purity is [among these] high and good silver dust of stability is desirable. It is also possible to mix an inorganic insulator in the range which may be mixed suitably, and may use these and does not lose conductivity. There is no limitation in particular in the form of a conductive filler, it can mix independently again and fillers, such as flaky, arborescence, and a bead type, can be used. As a commercial item, Tokuriki Chemical Research sill best TCG-1 (silver dust) etc. is mentioned. The amount of the conductive filler (C) used is usually the range of a 100 - 1000 weight part to a total amount 100 weight part with EPOSHIKI resin (A) and a curing agent (B).

[0014] Although there is no restriction in particular in the diluent (D) used for this invention, EPOSHIKI resin, What has the boiling point which is excellent in compatibility with a resultant with the poly dimethylsiloxane compound which has aliphatic monocarboxylic acid and a carboxyl group, a curing agent, etc., and does not volatilize to some extent easily from the workability of a resin paste is desirable. As such a diluent, for example Butyl cellosolve, butylcellosolve acetate, Carbitol, Carbitol acetate, ethylene glycol diethylether, Ethylene glycol dibutyl ether, butyl carbitol, butyl Carbitol acetate, Organic solvents, such as alpha-terpineol and diacetone alcohol, phenyl glycidyl ether, The monoglycidyl ether which has reactivity, such as p-tert-buthylphenyl glycidyl ether and p-sec-buthylphenyl glycidyl ether, KBM-403, LS-7970 (trade name by the Shin-etsu chemistry company), two-stage-liquefaction-8350, two-stageliquefaction-8355, two-stage-liquefaction-9905 (Toshiba Silicone trade name), The reactive diluent which has 1-2 epoxy groups is mentioned into 1 molecules, such as PGE (trade name by Nippon Kayaku Co., Ltd.), PP101 (trade name by Tohto Kasei Co., Ltd.), YED-122 (trade name by an oil recovery shell epoxy company), ED-502, and ED-503 (trade name by the Asahi electrification company). These are independent, or are combined two or more sorts and used. The amount of the diluent (D) used is usually a 0 - 800 weight part to the total amount 100 weight part of an epoxy resin (A) and a curing agent (B).

[0015] Adhesive strength improvers, such as a silane coupling agent and a titanium coupling agent, the Nonion system surface active agent, a fluorochemical surfactant, silicon oil, etc. are damp in the conductive resin paste of this invention, and an improver, a defoaming agent, etc. can be suitably added to it.

[0016] The epoxy resin with which the conductive resin paste of this invention has two or more epoxy groups in 1 molecule, for example, The resultant of the poly dimethylsiloxane compound

which has aliphatic monocarboxylic acid and a carboxyl group, The epoxy resin which has two or more epoxy groups in 1 molecule if needed, while adding heat-treatment for a diluent (D) if needed a curing agent (B) and if needed that it is simultaneous or separately -- churning, the dissolution, or **** -- it is made to mix and distribute, and it mixes and agitates, these mixtures are added a conductive filler (C) and distributed, and it is obtained. Although there is no restriction in particular in equipment, such as mixing, the dissolution, and dispersion, a flask reaction vessel, a RAIKAI machine, 3 rolls, a ball mill, a planetary mixer, etc. which were equipped with churning equipment and heating apparatus, for example can be used, and these may be combined suitably.

[0017] In order to paste up a semiconductor device on substrates, such as a lead frame, using the conductive resin paste of this invention After applying a conductive resin paste by the dispensing method, screen printing, the Stamping method, etc. on a substrate first, a semiconductor device is stuck by pressure and it can carry out by carrying out heating hardening using heating apparatus, such as oven or a heat block, after that. Furthermore, after passing through a wire bond process, it can be considered as the completed semiconductor device by closing by the usual method.

[0018]

[Example] An example explains this invention in detail hereafter.

Add poly dimethylsiloxane compound XC96-A2400 (trade name by Toshiba Chemical CORP.) 2.4g which has a carboxyl group to example 13 organic-functions epoxy resin TACTIX-742 (trade name by the Dow Chemical Co.) 50g, and 8.0g of caprylic acid (reagent), and it agitates at 120 degrees C. When it sampled for every definite period of time and the conversion by the acid number measurement by a potassium hydroxide alcoholic solution became 95% or more, it is a room temperature. It cooled, and butyl-cellosolve acetate (reagent) 50g was added to this, it heated and agitated at 50 degrees C, and the epoxy resin solution was obtained. To 30g of this epoxy resin, they are novolak phenol resin H-1 (Meiwa Chemicals trade name)60g and butyl-cellosolve acetate 40g. adding 12.0g of phenol resin solutions which carried out the heating dissolution at 100 degrees C -- further -- a sill -- the best -- TCG-1 (silver dust --) 95g of Tokuriki Chemical Research trade names and cure ZORU 2PHZ (imidazole, the Shikoku Chemicals trade name) 1.0g were added, it kneaded using a RAIKAI machine and 3 rolls, and the uniform conductive resin paste was obtained. The characteristics (viscosity, volume resistivity, a room temperature and the bonding strength at 350 degrees C, curvature of a chip) of the obtained conductive resin paste are investigated, and the result is shown in Table 1. Except having considered it as the combination shown in Table 1 in an example 2-7 and one to comparative example 3 example 1, each conductive resin paste is obtained like an example 1. and the result of a measurement of the characteristics is shown in Table 1. [0019]

[Table 1]

[Table 1]																		
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[0020] <The measuring method of characteristics> (1) viscosity: Using the EHD type rotational viscometer by Tokyo Keiki Co., Ltd., the 3-degree cone rotor was set and the measured value in 25 degrees C and 0.5rpm was acquired.

(2) Volume resistivity: the conductive resin paste was applied so that it might be set to 2mm in width, and 0.1mm in thickness on slide glass, heating hardening of this was carried out in 180-degree C oven for 1 hour, resistance was measured using the Wheatstone bridge, and volume resistivity rhov was computed by the following formula.

[Equation 1]

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ρ、(Ω・cm) = R・W・d/L
R:実測抵抗値(Ω)
W:サンプルの幅(cm)
d:サンプルの厚さ(cm)
L:測定端子間距離(cm)
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- (3) Bonding strength: the conductive resin paste was made to use and stick by pressure on copper flame with silver plating, heating hardening was carried out for 2 minutes in the 200-degree C heat block, and Si-chip cut to 2x2mm was used as the specimen. This room temperature and the shear bond strength at the time of 350-degree-C heating were measured using the push pull gauge (made by the Aikoh engineering company).
- (4) Curvature of a chip: when making a conductive resin paste use and stick by pressure on copper flame with silver plating and pasting up the silicon chip cut to 5x13mm in 180-degree C oven for 1 hour About ** chip curvature, it is the surface roughness meter DEKTAK. It measured in the scanning distance of 11mm using II (made in Sloane (SLOAN)). It is shown that the conductive resin paste of this invention is excellent in conductivity, workability, and bonding strength, and there is little chip curvature and it is excellent in the relaxation effect of thermal stress from Table 1.

[0021]

[Effect of the Invention] Since the thermal stress produced from the difference in the coefficient of thermal expansion between a semiconductor device and substrates, such as a lead frame, can be eased according to the conductive resin paste of this invention, The poor characteristics of the semiconductor device by the chip crack at the time of the heat-treatment especially in a semiconductor device assembly process or stress distortion can be prevented. Moreover, since it excels also in workability, hardenability, and an adhesive property, in the conventional conductive resin paste, the short-time hardening treatment of the difficult large-sized chip of application is attained.

[Translation done.]